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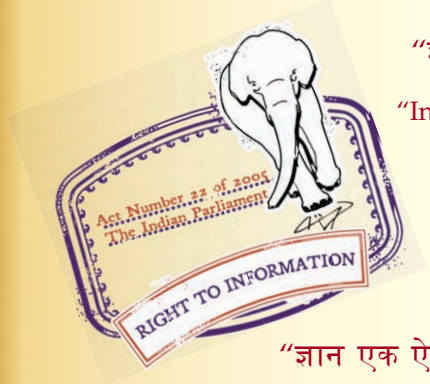
IS 3401 (1992): Silica gel [CHD 1: Inorganic Chemicals]



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IS 3401 : 1992

भारतीय मानक

(Reaffirmed 2003)

सिलिका जेल — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

SILICA GEL — SPECIFICATION

(Third Revision)

UDC 661·183·7

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1992

Price Group 3

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Devision Council.

Silica gel is pure silica in a highly porous state. It has high adsorption capacity due to the large surface area of its particles. Silica gel is normally prepared by the coagulation of colloidal solution of silicic acid.

Silica gel is used as a desiccant for packages. Its uses also cover prevention of corrosion in instruments, maintenance of dry atmosphere in food and pharmaceutical packings, removal of moisture from transformer breathers, protection of articles like cameras, televisions, etc, in humid atmosphere.

Silica gel is also used for adsorbing acetylene from oxygen gas and in chromatography for analytical purposes. However, this standard does not cover the material intended for these applications.

This standard was first published in 1966 and was based on IMD/SL/7061 (b) 'Desiccant silica', issued in 1957 by the Ministry of Defence, Govt of India. The standard was revised in 1970 incorporating changes in some of the requirements and the methods of test. It was again revised in 1979 incorporating changes in the requirements for moisture, chloride content for indicating type of silica gel, cobalt and sulphate content.

In this revision, changes have been made in description, loss on drying and adsorption capacity. The requirement for friability has been deleted and in its place a new requirement for loss on attrition has been incorporated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this standard.

Indian Standard

SILICA GEL — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for silica gel used as industrial desiccant.

1.1.1 It does not cover silica gel for chromatographic and acetylene adsorption applications.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this Standard:

IS No.	*Title*
460 : 1985	Test sieves (*third revision*)
695 : 1986	Acetic acid (*third revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
4161 : 1967	Nessler cylinders
4905 : 1968	Methods for random sampling

3 TYPES

The material shall be of following two types :

a) Normal, and

b) Indicating.

4 REQUIREMENTS

4.1 Description

The material shall be solid with grainy porous structure free from extraneous impurities. The indicating type material shall be impregnated with cobalt chloride.

4.2 Particle Size

The particle size of the material shall be as agreed to between the purchaser and the supplier.

4.3 Bulk Density

The bulk density of the material shall be as agreed to between the purchaser and the supplier.

4.4 Loss on Drying

The material when tested according to the method prescribed in Annex A shall not lose more than 5 percent of its mass.

4.5 Adsorption Capacity

The material when tested according to the method prescribed in Annex B shall adsorb minimum 27 percent of moisture on the basis of its mass.

4.6 *p*H

The *p*H of aqueous extract of the material when determined by the method prescribed in Annex C shall be not more than 8 and not less than 3.5.

4.7 Loss on Attrition

When subjected to the test according to the method prescribed in Annex D, not more than 2·5 percent of the material shall pass through the test sieve.

4.8 Chemical Requirements

The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Annex E. Reference to the relevant clauses of Annex E is given in col 4 of the table.

4.9 Keeping Quality

The material stored in original air-tight containers shall continue to satisfy all the requirements under **4** for not less than 6 months from the date of packing.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, dry and air-tight containers, as agreed to between the purchaser and the supplier.

Table 1 Requirements for Silica Gel

(*Clauses* 4.8 *and* E-5.3.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Annex E
(1)	(2)	(3)	(4)
i)	a) Chlorides (as NaCl) (for normal type), percent by mass, *Max*	0·05	E-3
	b) Chlorides (as NaCl) (for indicating type), percent by mass, *Max*	0·4	E-3
ii)	Cobalt (as $COCl_2$) (for indicating type), percent by mass, *Min*	0·5	E-4
iii)	Ammonium compounds (as NH_3), percent by mass, *Max*	0·001	E-5
iv)	Sulphates (as Na_2SO_4), percent by mass, *Max*	0·5	E-6

5.1.1 The container shall not be opened until required for use and shall not remain open for a period longer than required for taking out the material.

5.2 Marking

The containers shall be marked with the following information:

a) Name and type of the material,

b) Indication of the source of manufacturer,

c) Mass of the material in the container,

d) Batch number, and

e) Date of packing.

5.2.1 The containers may also be marked with the Standard Mark.

6 SAMPLING

The method of drawing representative samples of the material, the number of tests to be performed, and the criteria for conformity of the material to the requirements of the specification shall be as prescribed in Annex F.

ANNEX A
(*Clause* 4.4)

DETERMINATION OF LOSS ON DRYING

A-1 PROCEDURE

A-1.1 Weigh accurately about 30 g of the material in a flat-bottomed glass dish with ground-glass lid and keep it (after removing the lid) in an air-oven at 150 ± 5°C for 4 hours. Cool the dish and the lid to room temperature in a desiccator and weigh. Repeat the operation till constant mass is obtained.

A-2 CALCULATION

$$\text{Loss on drying, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = loss in mass in g of the material on heating, and

M = mass in g of the material taken for the test.

ANNEX B
(*Clause* 4.5)

DETERMINATION OF ADSORPTION CAPACITY

B-1 APPARATUS

B-1.1 The apparatus required shall be as shown in Fig. 1. It comprises the following:

a) Circulating pump with flowmeter.

b) Heat exchange coil, of sufficient length and efficiency to ensure that air leaving the pump is brought to the bath temperature before entering the bubbler.

c) Bubbler of about 250-ml capacity, fitted with a thermometer and containing a saturated solution of calcium nitrate of analytical reagent grade in the presence of excess solid calcium nitrate.

d) Spray-catch bottle of about 250-ml capacity, containing glass wool filter.

e) Tube with side arm, holding a thermometer for checking the temperature of air entering the adsorption tubes.

f) Two adsorption tubes, about 15 cm long and of 14 mm internal diameter.

g) Constant temperature bath, maintained at 27 ± 1°C.

NOTE — The underwater connections are conveniently made by means of flexible rubber or plastics tubing or spherical joints.

B-2 PROCEDURE

B-2.1 Weigh accurately about 10 g of the dry material immediately after the determination of loss on drying (*see* **A-1.1**) into each of the two previously weighed and stoppered adsorption tubes. Assemble the apparatus as shown in Fig. 1 and adjust the pump so that air is passed at a rate of approximately 1 litre per tube per minute. Allow air to circulate until no further increase in mass of the tubes is observed.

B-3 CALCULATION

$$\text{Adsorption capacity, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = increase in mass in g of the material, and

M = mass in g of the material taken for the test.

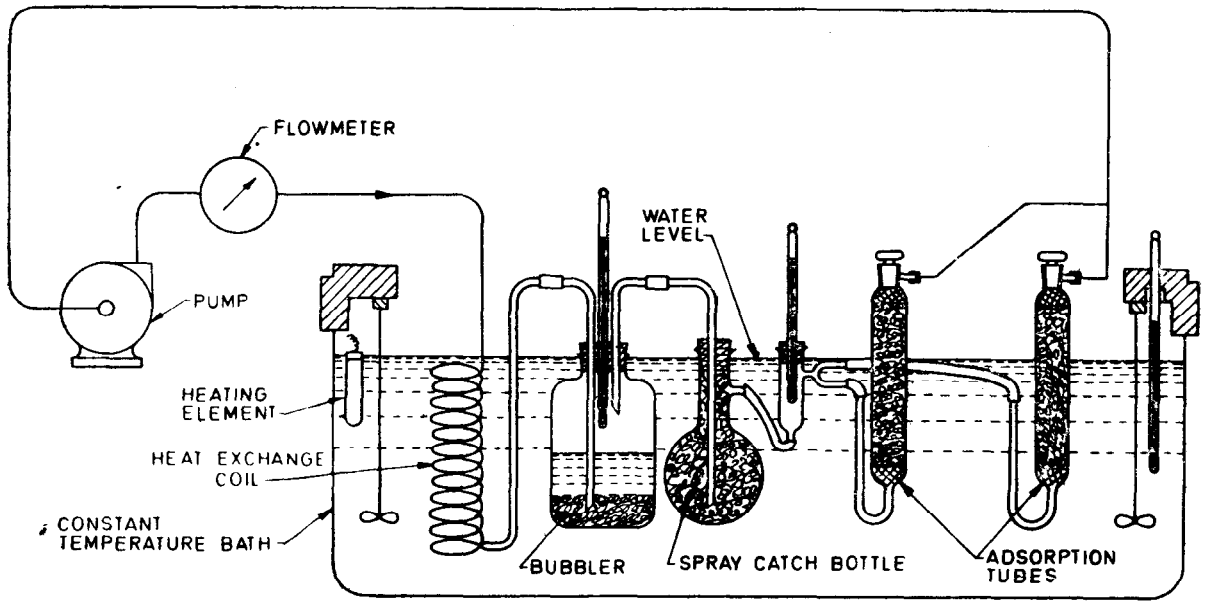

FIG. 1 APPARATUS FOR DETERMINATION OF ADSORPTION CAPACITY

ANNEX C

(*Clause* 4.6)

DETERMINATION OF *p*H

C-1 PROCEDURE

C-1.1 Prepare an aqueous extract of 25 g of the material by boiling for 2 hours with 250 ml of water, free from ammonia and carbon dioxide, in a clean flask of chemically resistant glass fitted by means of a ground-glass joint with a reflux condenser of the same material.

C-1.1.1 Allow the flask to cool to room temperature with adequate precautions against contamination by atmospheric impurities. Decant a portion of this extract and determine the *p*H by a suitable *p*H meter using a glass electrode previously calibrated with a buffer solution of known *p*H.

ANNEX D

(*Clause* 4.7)

DETERMINATION OF LOSS ON ATTRITION

D-1 PROCEDURE

D-1.1 Weigh accurately 50 g of the material as received or with 95 percent relative humidity. Charge the material into two steel cylinders of 3·8 cm diameter and 30·5 cm length. Rotate the cylinders kept at a distance of 7·6 cm from the centre of rotation (to enable the material to tumble) at 30 rpm for 1 h. Sieve the material through 850 micron IS Sieve (*see* IS 460 : 1985). Weigh the material passing through 850 micron IS Sieve and report as attrition loss.

D-1.1.1 The material shall satisfy the requirement of the test if not more than 2·5 g of the material pass through the test sieve.

ANNEX E
(*Clause* 4.8, and *Table* 1)

METHODS OF TEST FOR CHEMICAL REQUIREMENTS OF SILICA GEL

E-1 QUALITY OF REAGENTS

E-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

E-2 PREPARATION OF SOLUTION

E-2.1 Procedure

Weigh accurately about 25 g of the material and heat to boiling with 200 ml of water for 30 minutes. Filter and wash the residue with warm water till the washings are free from chlorides and sulphates. Mix the washings and make up to 500 ml in a volumetric flask. Use an aliquot of this prepared solution for subsequent tests under **E-3**, **E-4**, **E-5** and **E-6**.

E-3 DETERMINATION OF WATER SOLUBLE CHLORIDES

E-3.1 Reagents

E-3.1.1 *Standard Silver Nitrate Solution* — 0·1 N.

E-3.1.2 *Potassium Chromate Indicator Solution* — 5 percent (*m*/*v*).

E-3.1.3 *Dilute Acetic Acid* — 10 percent (*v*/*v*).

E-3.1.4 *Sodium Acetate Solution* — 10 percent (*m*/*v*).

E-3.2 Procedure

Take 200 ml of the prepared solution (*see* **E-2.1**), neutralise with sodium acetate solution in case the solution is of high acetic *p*H or neutralise with dilute acetic acid if the solution is of alkaline *p*H. Titrate it with 0·1 N silver nitrate solution using potassium chromate solution as indicator.

E-3.3 Calculation

Water soluble chlorides (as NaCl), percent by mass

$$= \frac{V \times N \times 0{\cdot}058\,45 \times 100}{M}$$

where

V = volume in ml of standard silver nitrate solution,

N = normality of standard silver nitrate solution, and

M = mass in g of the material contained in the aliquot.

E-4 DETERMINATION OF COBALT

E-4.1 Reagents

E-4.1.1 *Dilute Hydrochloric Acid* — 5 N.

E-4.1.2 *α-Nitroso β-Naphthol Solution*

Dissolve 3 g of dry α-nitroso β-naphthol in 45 ml of glacial acetic acid (*see* IS 695 : 1986).

E-4.2 Procedure

Take 50 ml aliquot of the prepared solution (*see* **E-2.1**). Dilute it with water to 400 ml, acidify with 10 ml of dilute hydrochloric acid and heat to boiling. Add 33 ml of α-nitroso β-naphthol solution with stirring. Allow to settle for 2 hours and filter through Whatman filter paper No. 41 or equivalent. Wash the residue with dilute hydrochloric acid and then thoroughly with hot water. Take the filter paper and the residue in a tared porcelain crucible and ignite first gently on the flame of a Bunsen burner and then finally at 750°C to 800°C. Cool in a desiccator and weigh to constant mass.

E-4.3 Calculation

Cobalt (as $CoCl_2$), percent by mass

$$= \frac{M_1 \times 1{\cdot}618 \times 100}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material present in the aliquot.

E-5 DETERMINATION OF AMMONIUM COMPOUNDS

E-5.1 Apparatus

E-5.1.1 *Nessler Cylinders* — 50-ml capacity (*see* IS 4161 : 1967).

E-5.2 Reagents

E-5.2.1 *Sodium Hydroxide Solution* — approximately 5 N.

E-5.2.2 *Nessler Solution*

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly with stirring saturated mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free

water. Allow to settle overnight, decant off the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

E-5.2.3 *Standard Ammonium Chloride Solution*

Dissolve 0·314 1 g of ammonium chloride in water and make up to 1 000 ml. Take 10 ml of the standard solution and dilute to exactly 100 ml with water. One millilitre of this solution is equivalent to 0·01 mg of ammonia (as NH_3).

E-5.3 Procedure

Take 20 ml of prepared solution (*see* **E-2.1**) into a Nessler cylinder. Add 5 ml of sodium hydroxide solution and 2 ml of Nessler solution. Make up the solution to 50-ml mark. Carry out a control test in another Nessler cylinder with 1 ml of standard ammonium chloride solution in place of the material and the same quantities of other reagents. Compare the colour produced in the two cylinders.

E-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

E-6 DETERMINATION OF WATER SOLUBLE SULPHATES

E-6.1 Reagents

E-6.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

E-6.1.2 *Barium Chloride Solution* — approximately 12 percent (*m/v*).

E-6.2 Procedure

Take 200 ml of the prepared solution (*see* **E-2.1**). Neutralize the excess alkali, if any, by dropwise addition of dilute hydrochloric acid and then add 2 ml more of dilute hydrochloric acid. Boil and add 10 ml of barium chloride solution slowly with constant stirring. Boil it for another 5 minutes and allow to settle overnight. Filter through Whatman filter paper No. 42 or equivalent and wash the residue with hot water till washings are free from chlorides. Ignite the residue in a tared crucible. Cool, add a drop of concentrated sulphuric acid and cautiously ignite again. Cool the crucible in a desiccator and weigh. Repeat the operation till constant mass is obtained.

E-6.3 Calculation

Water soluble sulphates (as Na_2SO_4), percent by mass

$$= \frac{M_1 \times 0{\cdot}608\ 6 \times 100}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material contained in the aliquot.

ANNEX F
(*Clause* 6)

SAMPLING OF SILICA GEL

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.1 The sampled material shall be kept in a protected place and shall not be exposed to damp air.

F-1.2 The sample shall be placed in suitable containers and each container shall be marked with full details of sampling giving the date of sampling; type of material; batch number, if any; and the name of the manufacturer.

F-2 SCALE OF SAMPLING

F-2.1 All the containers in a single consignment of silica gel of the same type from a single batch of manufacture shall constitute a lot.

F-2.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

F-2.3 The number of container (*n*) to be chosen from the lot shall depend on the size of the lot (*N*) and shall be as given in Table 2.

Table 2 Scale of Sampling

(*Clause* F-2.3)

Lot Size *N* (1)	No. of Containers to be Selected *n* (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 200	6
201 and above	8

F-2.4 The containers to be selected for sampling shall be chosen at random from the lot. For this purpose a random number table shall be used (*see* IS 4905 : 1968). In case such a table is not available, the following procedure shall be adopted:

Starting from any container, count them as 1, 2, 3, ..., *r*, and so on in a systematic manner, where *r* is the integral part of N/n. Every *r*th container thus counted shall be withdrawn to constitute the required sample.

F-3 TEST SAMPLE AND REFEREE SAMPLE

F-3.1 From each of the containers selected, draw approximately 150 g of the material with the help of suitable sampling implement. The material drawn from different containers shall be mixed thoroughly to give a composite sample weighing about 500 g.

F-3.2 The composite sample shall then be divided into three parts, one for the purchaser, another for the supplier and the third for the referee. These parts shall be transferred to separate containers which shall be suitably closed and marked with all the details of sampling.

F-3.3 The referee sample shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute.

F-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

F-4.1 Tests for all the characteristics given in **4** shall be conducted on the composite sample.

F-4.2 The lot shall be declared as conforming to the requirements of the specification if all the test results on the composite sample satisfy the relevant requirements given in **4**.

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TO
IS 3401 : 1992 SILICA GEL — SPECIFICATION

(Third Revision)

(*Page* 1, *clause* **4.8**, *Table* 1) — Substitute the following for existing table 1:

Table 1 Requirements for Silica Gel
(*Clause* **4.8** *and* **E-5.3.1)**

Sl No.	Characteristic	Requirement	Method of Test, Ref. to Cl No. in Annex E
(1)		(3)	(4)
i)	a) Chlorides (as NaCl) (for normal type), percent by mass, *Max*	0.05	E-3
	b) Chlorides (as NaCl) (for indicating type), percent by mass, *Max*	Chlorides (as NaCl) Equivalent of cobalt actually found in the sample +0.05	E-3
ii)	Cobalt (as $CoCl_2$) (for indicating type), percent by mass, *Min*	0.5	E-4
iii)	Ammonium compounds (as NH_3), percent by mass, *Max*	0.001	E-5
iv)	Sulphate (as Na_2SO_4) percent by mass, *Max*	0.5	E-6

(CHD 01)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JULY 2003
TO
IS 3401 : 1992 SILICA GEL — SPECIFICATION

(Third Revision)

(*Page* 4, *clause* **E-3.3**, *formula*) — Substitute '0.058 44' *for* '0.058 45'.

(*Page* 4, *clause* **E-4.3**, *formula*) — Substitute '1.617 5' *for* '1.618'.

(CHD 1)

Reprography Unit, BIS, New Delhi, India